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# **CANADIAN PATENT**

⑤④

**METHOD FOR FRACTIONATING SULFITE WASTE LIQUOR**

⑦⑩

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②①

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ABSTRACT OF THE DISCLOSURE

Disclosed is a method for multistep fractionation and concentration of sulfite waste liquor that has previously been stripped of free  $\text{SO}_2$ , cooled down and freed of fibres by filtration. The method combines ultrafiltration and reversed osmosis wherein fractions are concentrated and retained on membranes of successively finer porosity relative their molecular weights and washed as desired to remove residual low molecular components.

When wood chips and other cellulose containing, vegetable materials are digested according to the sulfite or bisulfite method a waste liquor appears which contains the water soluble contents of the raw material and quantitatively amounts to approximately half of the raw material. The composition of the sulfite waste liquor varies according to the used raw material, base, digesting temperature and time and the desired cellulose quality.

Numerous suggestions have been made as to the utilization of the sulfite waste liquor tapped from the digester, either in an evaporated or dried state, with or without its sugar content, which in many cases is removed by fermentation or oxidation.

According to a method for fractionating sulfite waste liquor known from US Specification No. 2 838 483 the lignosulfonic acids are absorbed on chrome tanned hide acidified with  $\text{SO}_2$ -water, undesirable components are washed out with water and the lignosulfonic acids are then released with ammonia. A disadvantage of this method is that it involves a large consummation of chemicals, and does not form a basis of a selective choice of molecule fractions. Also, the lignosulfonic acids are obtained in a concentration of only approximately 7%, which results in great costs in connection with concentration and drying. Furthermore, any utilization of the sugars present in the filtrate is made difficult, partly due to their low concentration and partly due to pollution with salts and traces of chrome. The preparation of the finely divided hide is also complicated and expensive.

The present invention has for its object to provide a simple and economic method for fractionating sulfite waste



liquor, so as to separate the main components of the waste liquor into high molecular lignosulfonate, low molecular lignosulfonate, sugar, salts and water in such a manner that each fraction is obtained in a suitable state for further use as regards concentration and composition.

10 The invention thus relates to a method of separating by reverse osmosis high and low molecular weight lignosulfonates from waste sulfite liquor and from each other, the liquor having previously been stripped of free  $\text{SO}_2$ , cooled down and freed of fiber by filtration, comprising passing the liquor in a stream over the primary side of a semi-permeable membrane under high pressure to retain the high molecular weight lignosulfonates in a first part of the stream on the primary side and pass a second part of the liquid stream with the low molecular weight lignosulfonates through the membrane to the secondary side; preparing a mixture of the high molecular weight first part stream with fresh liquor, recirculating the mixture over the primary side to concentrate the high molecular weight lignosulfonates in the first part of the stream on the primary side and provide additional  
20 second parts of the stream with low molecular weight lignosulfonates on the secondary side; and recovering all the second parts with the low molecular weight lignosulfonates from the secondary side of the membrane.

The waste liquor is thus forced to circulate from the liquor tank via a pump, through membranes and back to the liquor tank. At the delivery side of the membrane a pressure regulator is preferably arranged. In the circulation, i.e. within the apparatus, the operational pressure is up to approximately 15 atmospheres.

As compared with the mentioned US Specification No. 2 838 483 clear and considerable advantages are achieved by the present method, a product of improved quality being obtained, without the need of supplying chemicals involving discharge problems, and lignosulfonate being obtained in considerably larger concentrations, that is approximately 25 - 30% against approximately 7% according to US Specification No. 2 838 483. For a certain capacity there is also considerably less need of investment for production equipment.

10           The invention is now to be explained in detail with reference to the accompanying drawings, where the method is diagrammatically shown. Fig. 1 shows a fractionating unit, fig. 2 shows a battery for separating various fractions, and fig. 3 shows the means for washing the retained fractions.

          With reference to fig. 1 the sulfite waste liquor flows from the liquor tank 1 through the high pressure circulation pump 2 with high velocity over the membrane faces 3, whereby the concentrating polarisation of the membranes is reduced, and low molecular components and water pass through the membranes to their  
20   secondary side. Said secondary side communicates with atmosphere through the discharge pipe 4 for filtrate or permeate. Said discharge pipe runs to the filtrate tank 5 for collection for the next step treatment.

          The remaining portion of the sulfite waste liquor is led through a line 6 and the pressure regulator 7 back to the liquor tank. This fraction is enriched with high molecular components.

          The recirculation of this fraction is continued until concentration is so much increased that the viscosity and osmo-

tic pressure of the liquid is such that the filtration capacity is too low. This will take place when the recirculating liquid has a concentration of solids of 25 - 35%, corresponding to approximately 55 - 75% of the content of solids in the originally supplied liquor.

If desired, this fraction can be washed with water during another circulation through the apparatus by use of the same membranes during continuous water supply. The residual low molecular components will then be removed with the filtrate and the product yielded is pure high molecular lignosulfonate. The purity may be adjusted according to the washing time.

The filtrate as well as concentrated filtrate from the washing operation, discharged through the pipe 4, may in turn be concentrated and subjected to further fractionating to lignosulfonate with lower molecular weight in the same manner as stated above, but by use of finer membranes 3. The production of pure low molecular lignosulfonates is carried out in the same manner as described in connection with the high molecular components.

When high and low molecular lignosulfonates have been separated a liquid remains containing sugars and salts of the waste liquor components. These cannot be separated by membranes due to too small a difference in their molecular weight, but have to be separated chemically, or e.g. by prefermentation.

In Fig. 2 a battery is shown for a continuous process according to the invention. The separate parts have the same reference symbols as in Fig. 1 but with an added index. Thus, the membranes 3 are coarse-pored, the membranes 3a have medium porosity, and the membranes 3c are fine-pored. The filtrate tanks 5a and 5b respectively in their respective steps correspond to

the liquor tank 1 of Fig. 1.

In Fig. 3 10 is a module for ultrafiltration or reversed osmosis, 11 is a circulation tank, 12 a circulation pump, 13 a collecting pipe for permeate or filtrate discharge, 14 is a pressure regulator, 15 a level controlled valve for supply of washing water to the concentrate, 16 is a float for level control, 17 is a thermostat mixer for wash water for the concentrate, 18 a supply for cold water, 19 a supply for hot water, 20 a supply for liquor, filtrated and tempered, 21 a discharge for washed concentrate, 22 an insulation, 23 a supply for washing water to the membranes, 24 a discharge for washing water from the membranes, and 25 and 26 are valves.

The amount of liquor to be fractionated is introduced into the tank 11, which is insulated by a cover and an exterior insulation. In this circulation tank a float 16 is arranged for level control. The liquor leaves the tank 11 through the circulation pump 12 and is introduced into the arrangement or module 10 for ultrafiltration or reversed osmosis at the levels indicated by arrows. The liquor passing the membrane that is indicated by dotted lines, flows through the pressure regulator 14 back to the circulating tank 11, whereas the filtrate or permeate is removed through a collecting line 13 to be supplied to the tank for the next fractionating step.

When permeate leaves the unit 10 the level in the circulation tank 11 is reduced. When the level in this tank reaches the dotted line, the level valve 15 is opened for supply of washing water to the concentrate. The supplied washing water is tempered, partly consisting of hot water supplied through the pipe 19 and partly consisting of cold water supplied through the pipe 18. In the thermostat mixer 17 the water supply is tempered



to the desired temperature. During washing operations the level in the circulation tank is controlled by the float 16 and maintains the level indicated by the dotted line. The ready washed concentrate is tapped through the pipe 21, whereupon the tank 11 is filled with a new batch of liquor.

To clean the membranes washing water is supplied through the pipe 23, while valves 25 and 26 are closed. The washing water passes through the circulation 23, 12, 10, and 14 and is discharged through the pipe 24.

- 10           The following examples illustrate the invention, and reference is made to the table on page 20, illustrating the quantitative distribution of liquor solids in the various fractions of the following examples.

EXAMPLE 1

Used sulfite waste liquor: Ca-base liquor from paper pulp sulfite digestion of pine.

Concentration of solids : 15.0%

Pre-treatment : Stripped of free  $\text{SO}_2$ , cooled down to approximately  $20^\circ\text{C}$  and filtered off

- 20   Separating limit of :  
the used membrane : Molecular weight appr. 20,000

17.55 kg of the specified Ca sulfite waste liquor, containing 2630 g solids were circulated for 35 minutes. After completion of the test there were 2 different fractions showing the following specifications:

- I. Concentrated high molecular Ca-lignosulfonate with low molecular Ca-lignosulfonate, organic acids,  $\text{CaSO}_3$  and sugar in original concentration, in minor amounts as a ballast.

Amount: 7.55 kg, concentration: 26.2% solids.

- 30   Amount of solids: 1980 g, corresponding to 75.3% of supplied solids.

II. The dominating quantity of low molecular Ca-lignosulfonate, organic acids,  $\text{CaSO}_3$  and sugar, in original concentration.

Amount: 10.0 kg, concentration: 6.5% solids.

Amount of solids: 650 g, corresponding to 24.7% of the supplied solids.

Utilization of the two fractions:

Fraction I:

- a) Final concentration by evaporation and combustion for steam production.
- 10 b) Final concentration by evaporation and pulverization for pelletization, dispersion.
- c) Washing out of ballast (undesired components) for the production of a highly refined Ca-lignosulfonate for finer dispersing, precipitation of water soluble proteins in butcher's waste water etc. Described in Example 2.

Fraction II:

- a) Suitable raw material for prefermentation with proper pH-adjustment and preceding precipitation with  $\text{CaO}$ , resulting in the possibility of recovering  $\text{CaSO}_3$  to be returned to the preparation of digestion acid.
- 20 b) Concentration by reversed osmosis over the finest-pore membrane followed by final evaporation and combustion for steam production.

#### EXAMPLE 2

Used raw material

: Fraction I from example 1, i.e. concentrated high molecular Ca-lignosulfonate,, with low molecular Ca-lignosulfonate, organic acids,  $\text{CaSO}_3$  and sugar in original concentration,

in smaller quantities, as a ballast.

Separating limit of  
used membrane : molecular weight appr. 20,000.

7.4 kg of said substance, with a concentration of 26.2% of solids, corresponding to a content of solids of 1940 g were washed in the apparatus for 65 minutes as described in connection with fig. 3. After completion of the test there were 2 different fractions having the following specification:

- I. Concentrated high molecular Ca-lignosulfonate of a high degree of purity, suitable for demanding tasks.  
Amount : 5.25 kg, concentration 23.9% solids.  
Amount of solids: 1255 g, corresponding to 64.7% of the supplied solids, i.e. 48.7% of the originally supplied solids. (Example 1)
- II. Filtrate containing a smaller portion of the 10 molecular components of the liquor in a diluted state.  
Amount: 32.65 kg, concentration: 2.1% solids.  
Amount of solids: 685 g, corresponding to 35.3% of the supplied solids, i.e. 26.6% of the originally supplied solids. (Example 1)

Utilization of the two fractions:

#### Fraction I:

Final concentration by evaporation and pulverization for dispersion plasticizing (water reducing agent) and precipitation of water soluble proteins for the purification of sewage from butchers etc.

#### Fraction II.

Concentration by reversed osmosis over the finest-pore membrane retaining appr. 95% of the solids, with following

combination with fraction II from Example 1 for further treatment (Example 3).

### EXAMPLE 3

Used raw material : Fraction II from Example 1, i.e.  
appr. half of the quantity of low  
molecular Ca-lignosulfonate, organic  
acids,  $\text{CaSO}_3$  and sugar in original  
concentration

10 Separating limit of  
used membrane : Molecular weight appr. 50.

10.0 kg of said substance, having a concentration of  
6.5 kg solids, corresponding to a content of solids of 650 g,  
were circulated for 43 minutes. After completion of the test  
there were 2 different fractions having the following specifications:

I. A concentrated solution of low molecular Ca-lignosulfonate,  
organic acids,  $\text{CaSO}_3$  and sugar.

Amount: 3.96 kg, concentration: 15.6% solids.

20 Amount of solids: 618 g, corresponding to 95.2% of the  
supplied solids, i.e. 23.5% of the originally supplied  
solids. (Example 1)

II. Filtrate with very small amounts of the low molecular components.

Amount: 5.5 kg, concentration: 0.59% solids.

Amount of solids: 32 g, corresponding to 4.8% of the supplied  
solids, i.e. appr. 1.2% of the originally supplied solids.  
(Example 1).

Utilization of the two fractions:

Fraction I:

- a) Final concentration by evaporation and combustion for steam production.
- b) Raw material for the production of fermentation protein.
- Fraction II:

For sewage or re-use.

EXAMPLE 4

Used sulfite waste liquor

- type :  $\text{NH}_4$  base liquor from paper pulp sulfite digestion of pine
- 10 Concentration of solids : 11.5%
- Pretreatment : Stripped from free  $\text{SO}_2$ , cooled down to appr.  $20^\circ\text{C}$  and fibres filtered off.

Separating limit of

used membrane : Molecular weight appr. 20.000.

9.9 kg of the specified  $\text{NH}_4$  sulfite waste liquor, containing 1140 g solids were circulated for 15 minutes.

After completed test there were 2 different fractions having the following specification:

- 20 I. Concentrated high molecular  $\text{NH}_4$ -lignosulfonate, with low molecular  $\text{NH}_4$ -lignosulfonate, organic acids,  $(\text{NH}_4)_2\text{SO}_3$  and salts in original concentration, in minor quantities, as a ballast.

Amount: 2.6 kg, concentration: 26.9% solids.

Amount of solids: 700 g, corresponding to 61.5% of the supplied solids.

- II. The dominating quantity of low molecular  $\text{NH}_4$ -lignosulfonate, organic acids,  $(\text{NH}_4)_2\text{SO}_3$  and sugar, in original concentration.

30 Amount: 7.3 kg, concentration: 6.03% of solids.

Amount of solids: 440 g, corresponding to 38.5% of the supplied solids.

Utilization of the two fractions:

Fraction I:

- a) Final concentration by evaporation and combustion for steam production.
- b) Final concentration by evaporation and pulverization for pelleting, dispersion.
- c) Washing out of low molecular components for the production of pure high molecular  $\text{NH}_4$ -lignosulfonate as described before.

10 Fraction II:

- a) Suitable raw material for prefermentation, containing ammonia, which serves as a ferment nutrition in a suitable process of prefermentation.
- b) Concentration by reversed osmosis over the finest-pore membrane with following final evaporation and combustion for steam production.

#### EXAMPLE 5

Used raw material : Fraction I from Example 4, i.e. concentrated high molecular  $\text{NH}_4$ -lignosulfonate, with low molecular  $\text{NH}_4$ -lignosulfonate, organic acids,  $(\text{NH}_4)_2\text{SO}_3$  and sugar in original concentration, in minor quantities, as a ballast.

Separation limit of

the used membrane : Molecular weight appr. 20.000

2.6 kg of said substance, with concentration: 26.9% solids, corresponding to a content of solids of 700 g, were washed for 24 minutes. After completion of the test there were 2 different fractions having the following specifications:

30

I. Concentrated, high molecular  $\text{NH}_4$ -lignosulfonate of high purity.

Amount: 2.8 kg, concentration: 17.3% solids.

Amount of solids: 485 g, corresponding to 67.9% of the supplied solids, i.e. 42.6% of the originally supplied solids. (Example 4)

II. Filtrate containing a minor portion of the low molecular components of the waste liquor in a diluted state.

Amount: 15.2 kg, concentration: 1.42% solids.

10 Amount of solids: 215 g, corresponding to 32.1% of the supplied solids, i.e. 18.9% of the originally supplied solids. (Example 4)

Utilization of the two fractions:

Fraction I:

Directly, or via conversion with a base to metal salts of lignosulfonic acid, final concentration and pulverization for dispersion, plasticizing or precipitation of water soluble proteins from sewage water from butchers or the like. By use of a strong base a possibility of recovering ammonia to be separated by heating is achieved.

Fraction II:

Concentration by reversed osmosis over the finest-pore membrane retaining appr. 95% of the solids, with subsequent combination with fraction II from Example 4, for further treatment.

#### EXAMPLE 6

Used raw material : Fraction II from Example 4, i.e. the main portion of the low molecular  $\text{NH}_4$ -lignosulfonate, organic acids,

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( $\text{NH}_4$ ) $_2\text{SO}_3$ , and sugar, in original concentration.

Separation limit of the used membrane : Molecular weight appr. 50.

7.3 kg of said substance with a concentration of 6.03 % solids, corresponding to 440 g solids were recirculated for 50 minutes. After completion of test there were two different fractions having the following specifications:

I. A concentrated solution of low molecular  $\text{NH}_4$ -lignosulfonate, organic acids, ( $\text{NH}_4$ ) $_2\text{SO}_3$ , and sugar.

Amount: 2.58 kg, concentration: 16.3 % solids.

Amount of solids: 420 g, corresponding to 95.5 % of the supplied solids, i.e. 36.8 % of the originally supplied solids. (Example 4).

II. Filtrate (fraction II, Example 4) with very small amounts of the low molecular components.

Amount: 4.72 kg, concentration: 0.42 % solids,

Amount of solids: 20 g, corresponding to 4.5 % of the supplied solids, i.e. appr. 1.7 % of the originally supplied solids.

Utilization of the two fractions:

Fraction I:

a) Final concentration by evaporation and combustion for steam production.

b) Raw material for the production of fermentation protein.

Fraction II:

For sewage or re-use.

#### EXAMPLE 7

Used sulfite waste liquor; Mg-base liquor from sulfite digestion of pine.



Concentration of solids : 11.2%

Pretreatment : Stripped from free  $\text{SO}_2$ , cooled down to appr. 20°C and fibres filtered off.

Separation limit of the

used membrane : Molecular weight appr. 20.000.

18.3 kg of the specified Mg-sulfite waste liquor, containing 2050 g solids, were recirculated for 55 minutes.

After completion of test there were 2 different fractions

10 having the following specifications:

I. A concentrated high molecular Mg-lignosulfonate, with low molecular Mg-lignosulfonate, organic acids,  $\text{MgSO}_3$ , and sugar in original concentration as well.

Amount: 4.6 kg, concentration: 30.4% solids.

Amount of solids: 1400 g, corresponding to 68.4% of the supplied solids.

II. The dominating quantity of low molecular Mg-lignosulfonate, organic acids,  $\text{MgSO}_3$ , and sugar in original concentration.

Amount: 13.7 kg, concentration: 4.75% solids.

20 Amount of solids: 650 g, corresponding to 31.6% of the supplied solids.

Utilization of the two fractions:

Fraction I:

- a) Final concentration by evaporation and combustion for recovery of base.
- b) Washing out of low molecular components for the production of a more or less pure high molecular Mg-lignosulfonate for dispersion or as a binding agent for special objects.

Fraction II:

- 30 a) Concentration by reversed osmosis over the finest-pore membrane with subsequent final concentration by evaporation and combustion for recovery of base (Example 8)
- b) Prefermentation of the sugar content.

EXAMPLE 8

Used raw material : Fraction II from Example 7, a filtrate containing the dominating quantity of low molecular Mg-lignosulfonate, organic acids,  $\text{MgSO}_3$  and sugar in original concentration.

Separation limit of

the used membrane : Molecular weight appr. 50.

10 13.6 kg of said substance, containing 645 g solids in a 4.75% concentration were recirculated for 40 minutes. After completion of the test there were two different fractions having the following specifications:

I. A concentrated solution of low molecular Mg-lignosulfonate, organic acids,  $\text{MgSO}_3$ , and sugar.

Amount: 3.34 kg, concentration: 17.6% solids.

Amount of solids: 587 g, corresponding to 91.2% of the supplied solids, i.e. 28.9% of the originally supplied solids. (Example 7)

20 II. Filtrate (Example 7, fraction II) with very small amounts of low molecular components.

Amount: 9.9 kg, concentration: 0.59% solids.

Amount of solids: 58 g, corresponding to 8.8% of the supplied solids, i.e. appr. 2.7% of the originally supplied solids. (Example 7)

Utilization of the two fractions:

Fraction I:

Final concentration by evaporation and combustion for recovery of base.

30 Fraction II:

For sewage or re-use.

EXAMPLE 9

Used sulfite waste liquor : Ca-base liquor from paper pulp sulfite digestion of birch.

Concentration of solids : 13.5%

Pretreatment : Stripped of free  $\text{SO}_2$ , cooled down to 25°C and fibres filtered off.

Separation limit of the

used membrane : Molecular weight appr. 20.000.

15.0 kg of the specified Ca-sulfite waste liquor, containing 2025 g of solids, were recirculated for 40 minutes. After completion of the test there were two different fractions having the following specifications:

- 10 I. A concentrated high molecular Ca-lignosulfonate of birch with low molecular Ca-lignosulfonate, organic acids,  $\text{CaSO}_3$ , and sugar as well.  
Amount: 4.5 kg, concentration: 25.1% solids.  
Amount of solids: 1130 g, corresponding to 55.8% of the supplied solids.
- II. The dominating quantity of low molecular Ca-lignosulfonate, organic acids,  $\text{CaSO}_3$ , and sugar, in original concentration.  
Amount: 10.5 kg, concentration: 8.52% of solids.  
Amount of solids: 895 g, corresponding to 44.2% of the supplied solids.
- 20

Utilization of the two fractions:

Fraction I:

- a) Washing with water over the same membrane for the production of pure high molecular Ca-lignosulfonate of birch for the precipitation of proteins in sewage.  
Unsuitable as a dispersing agent.
- b) Final concentration by evaporation and combustion for steam production.

Fraction II:

- 30 a) A raw material for the production of ferment protein.  
b) A raw material for the production of pentose (xylose).  
c) Concentration over a finer membrane with subsequent evaporation and combustion for steam production.

- d) A raw material for the production of pure, low molecular Ca-lignosulfonate (Example 10).

EXAMPLE 10

Used raw material : Fraction II from Example 9, i.e. the dominating quantity of low molecular Ca-lignosulfonate, organic acids,  $\text{CaSO}_3$ , and sugar, in original concentration.

Separation limit of the

- 10 used membrane : Molecular weight appr. 1000.

10.4 kg of said substance with a concentration of 8.52% solids corresponding to a content of solids of 885 g were recirculated for 15 minutes. After completion of the test there were two different fractions having the following specifications:

- I. Concentrated low molecular Ca-lignosulfonate, with an amount of organic acids,  $\text{CaSO}_3$ , and sugar, in original concentration.

Amount: 3.5 kg, concentration: 15.6% solids.

- 20 Amount of solids: 545 g, corresponding to 61.6% of supplied solids, i.e. 27.1% of originally supplied solids. (Example 9)

- II. The main amount of organic acids,  $\text{CaSO}_3$ , and sugar in original concentration.

Amount: 6.9 kg, concentration: 4.9% solids.

Amount of solids: 340 g, corresponding to 38.4% of the supplied solids, i.e. 16.9% of originally supplied solids (Example 9).

Utilization of the two fractions:

- 30 Fraction I:

- a) Washing with water for the production of pure low molecular Ca-lignosulfonate for the precipitation of proteins. The filtrate from the washing process is concentrated by

reversed osmosis and combined with fraction II from Example 10.

Fraction II:

- a) A raw material for the production of ferment protein (torula).
- b) A raw material for the production of pentose (d-xylose).

#### EXAMPLE 11

10      Used raw material                    : Fraction II from Example 10, i.e. the main amount of organic acids,  $\text{CaSO}_3$ , and sugar, in original concentration.

Separation limit of the

used membrane                    : Molecular weight appr. 50.

6.9 kg of said substance, having a concentration of 4.9% of solids, corresponding to a content of solids of 340 g, were recirculated for 65 minutes. After completion of test there were two different fractions having the following specifications:

- I. Organic acids,  $\text{CaSO}_3$  and sugar concentrated.  
20      Amount: 3.2 kg, concentration: 10.4% of solids.  
Amount of solids: 332.6 g, corresponding to 97.8% of supplied solids, i.e. 16.6% of the originally supplied solids (Example 9).
- II. Filtrate containing very small amounts of the low molecular components of the liquor.

Utilization of the two fractions:

Fraction I:

A raw material for the production of pentose (xylose)

Fraction II:

30      For sewage or re-use.

The results are shown in the following table.

Fractioning of various sulfite waste liquors  
 Survey of distribution of solids among the  
 different fractions and concentrations in these

Waste liquor	Membrane separation limit	Int. solution			I. Concentrate			II. Filtrate			
		solids % conc.	solids grams	solids % conc.	solids grams	solids % of supply orig.	solids % conc.	solids grams	solids % of supply orig.	solids % conc.	solids grams
1. Ca-pine	m.w. 20.000	15.0	2630	26.2	1980	75.3	6.5	650	24.7	24.7	24.7
2. do.1./I.	m.w. 20.000	26.2	1940	23.9	1255	64.7	2.1	685	35.3	26.6	26.6
3. do.1./II.	m.w. 50	6.5	650	15.6	618	95.2	0.59	32	4.8	1.2	1.2
4. NH <sub>4</sub> -pine	m.w. 20.000	11.5	1140	26.9	700	61.5	6.03	440	38.5	38.5	38.5
5. do.4./I.	m.w. 20.000	26.9	700	17.3	485	67.9	1.42	215	32.1	18.9	18.9
6. do.4./II	m.w. 50	6.03	440	16.3	420	95.5	0.42	20	4.5	1.7	1.7
7. Mg-pine	m.w. 20.000	11.2	2050	30.4	1400	68.4	4.75	650	31.6	31.6	31.6
8. do.7./II	m.w. 50	4.75	645	17.6	587	91.2	0.59	58	8.8	2.7	2.7
9. Ca-birch	m.w. 20.000	13.5	2025	25.1	1130	55.8	8.52	895	44.2	44.2	44.2
10. do.9./II	m.w. 1.000	8.52	885	15.6	545	61.6	4.9	340	38.4	16.9	16.9
11. do.10./II	m.w. 50	4.9	340	10.4	333	97.8	0.2	7.4	2.2	0.36	0.36

The embodiments of the invention in which an exclusive property or privilege is claimed are defined as follows:

1. The method of separating by reverse osmosis high and low molecular weight lignosulfonates from waste sulfite liquor and from each other, the liquor having previously been stripped of free  $\text{SO}_2$ , cooled down and freed of fiber by  
5 filtration, comprising: passing said liquor in a stream over the primary side of a semi-permeable membrane under high pressure to retain said high molecular weight lignosulfonates in a first part of said stream on said primary side and pass a second part of said liquid stream with the low molecular weight lignosulfon-  
10 ates through the membrane to the secondary side; preparing a mixture of said high molecular weight first part stream with fresh said liquor; recirculating said mixture over said primary side to concentrate the high molecular weight lignosulfonates in the first part of said stream on the primary side and provide additional  
15 second parts of said stream with low molecular weight lignosulfonates on said secondary side; and recovering all said second parts with the low molecular weight lignosulfonates from said secondary side of the membrane.

2. The method of Claim 1 wherein said high pressure is maintained with a high pressure pump for said liquor upstream of said membrane.

3. The method of Claim 1 wherein said recirculation is continued until about 55-75% of the solids in said first part of the waste liquor have been removed.

4. The method of Claim 1 wherein said pressure is superatmospheric up to a pressure of about 15 atmospheres.

5. The method of Claim 1 wherein said membrane has a separating limit of about 20,000 molecular weight.

6. The method of Claim 1, Claim 3 or Claim 4 wherein

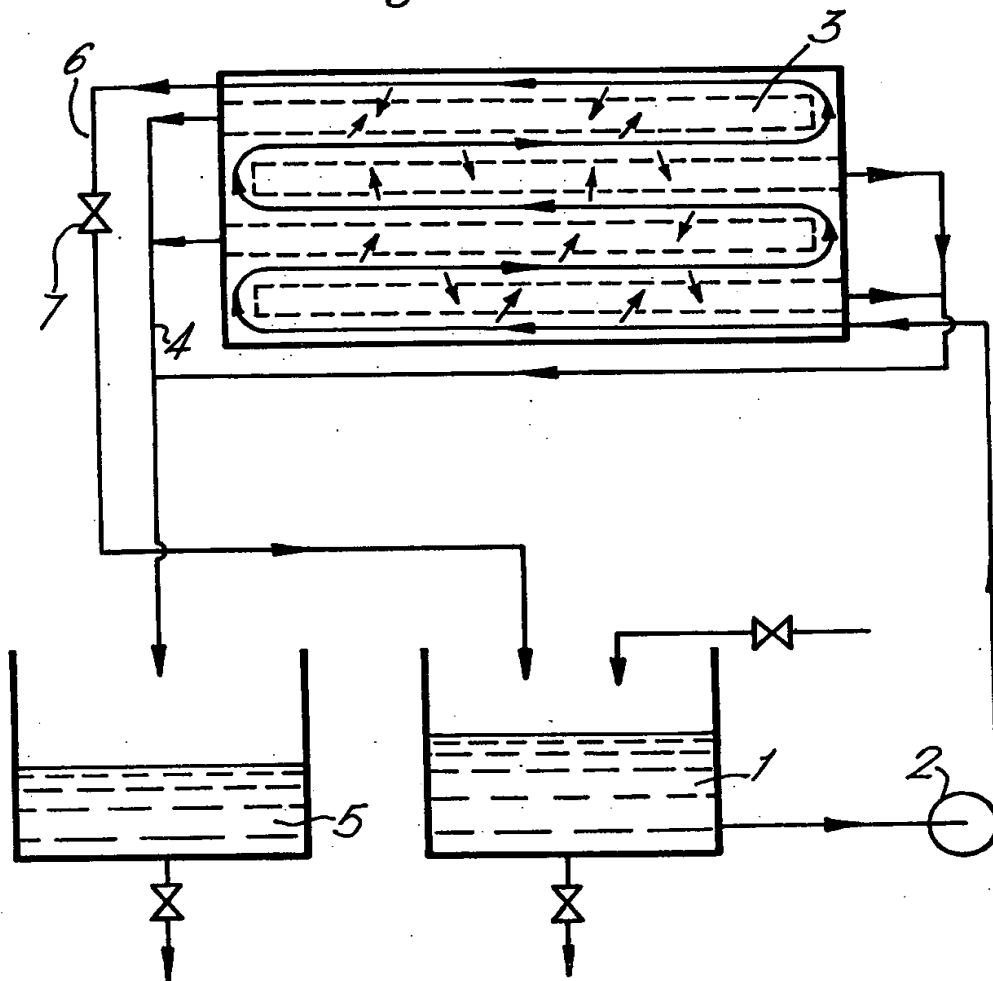
said first part of said stream is washed with water  
over said primary side thereby removing residual low molecular  
weight lignosulfonates with the resulting second stream part  
5 through the membrane and providing a high molecular weight  
lignosulfonate product on the primary side in a purified state  
which is dependent upon the amount of said washing.

7. The method of Claim 1, Claim 3 or Claim 4 wherein  
said second part of said liquor with the low molecular weight  
lignosulfonates is subjected to said reverse osmosis separation  
through successively finer membranes to fractionate the sul-  
5 fonates into progressively lower molecular weight fractions.





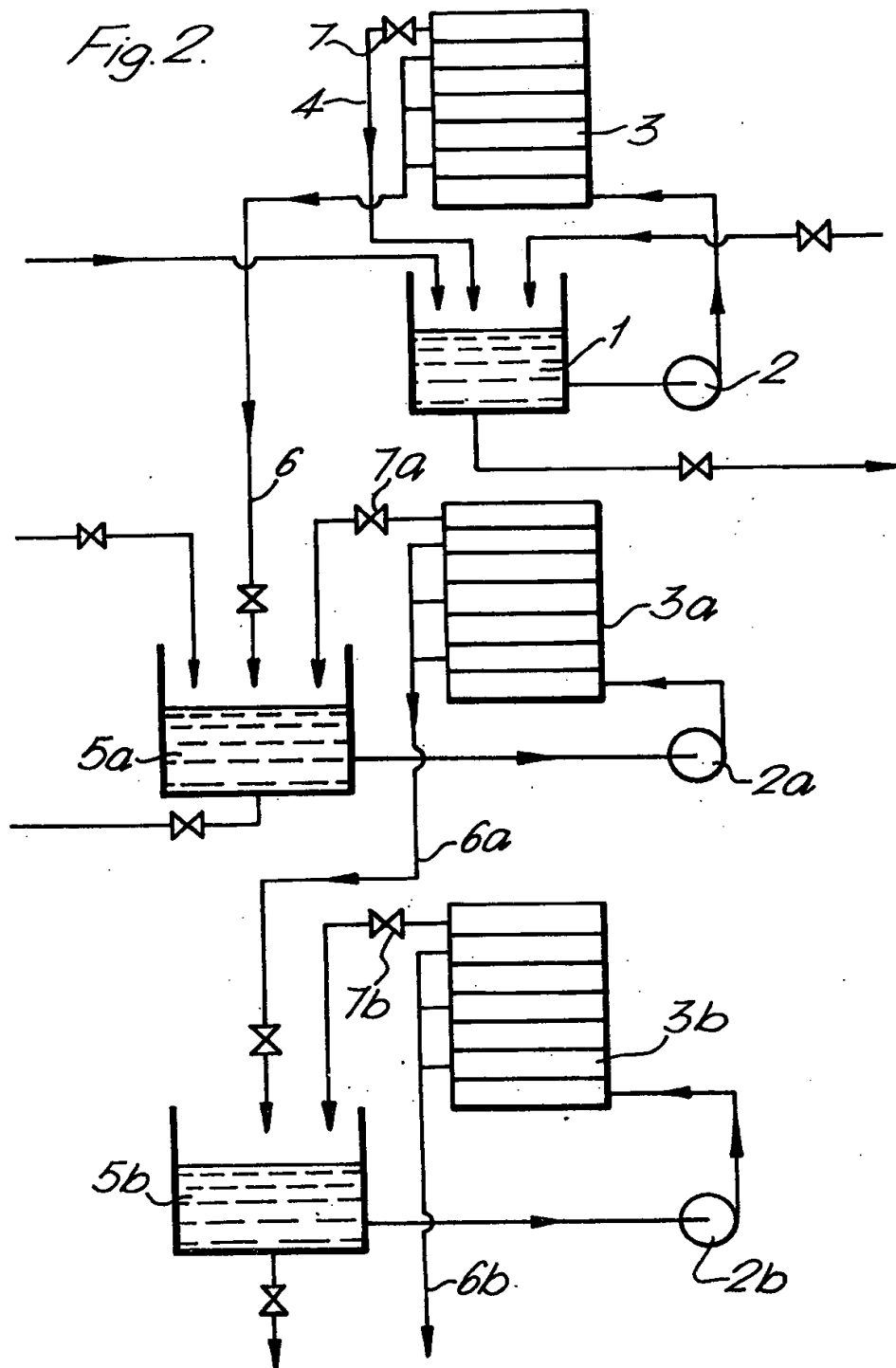
Fig. 1.



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Fig. 2.



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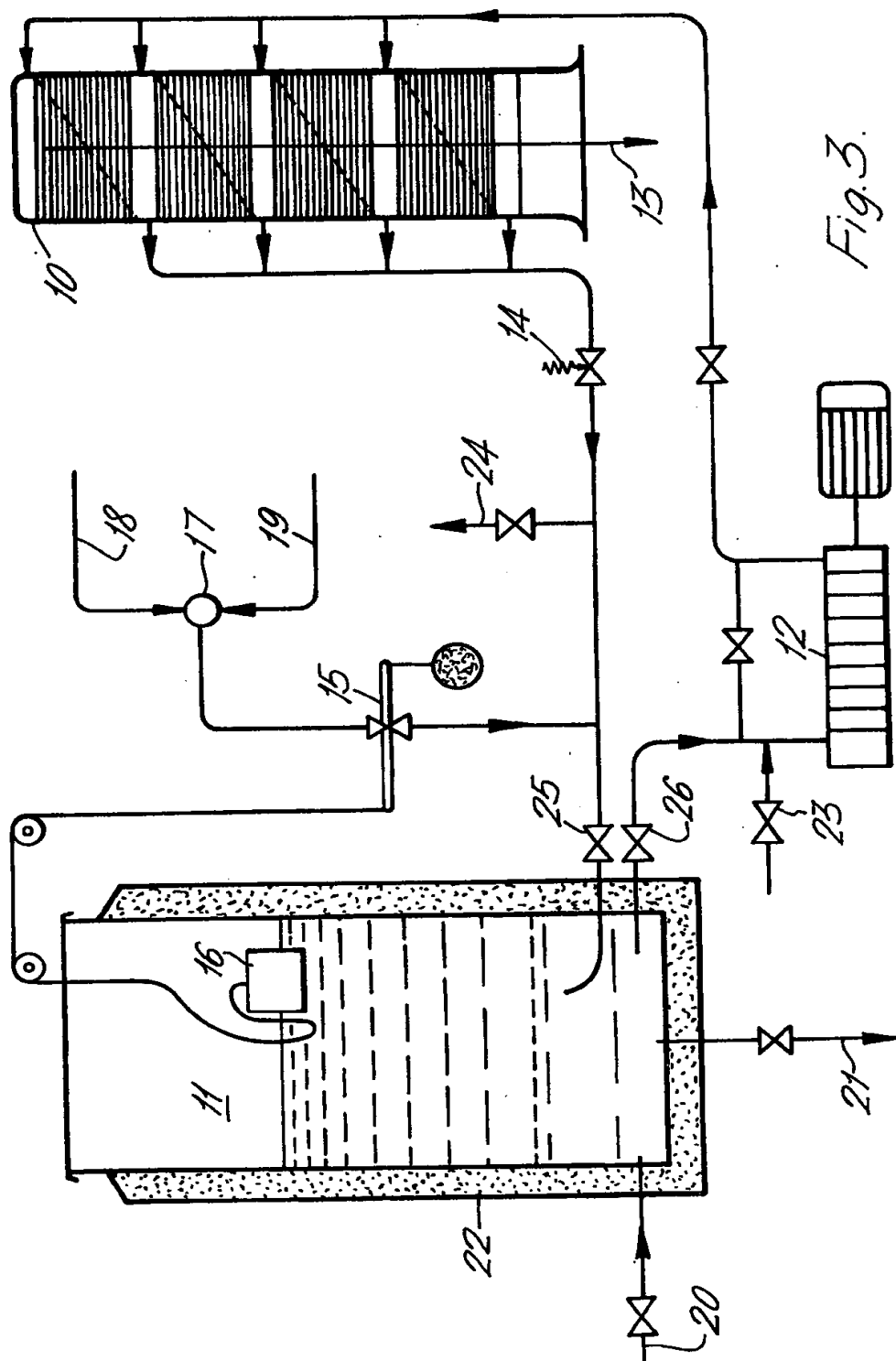


Fig. 3.

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